भारतीय मानक **IS 5282 (Part 1) : 2023**

Indian Standard

गुरुत्वाकर्षण तरल अवसादन विधियों द्वारा कण आकार वितरण का निर्धारण

भाग 1 सामान्य सिद्धांत और दिशानिर्देश

(पहला पुनरीक्षण)

Determination of Particle Size Distribution by Gravitational Liquid Sedimentation Methods

Part 1 General Principles and Guidelines

(First Revision)

ICS 19.120

© BIS 2023

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

August 2023 **Price Group 7**

Sieves, Sieving and Other Sizing Methods Sectional Committee, CED 55

FOREWORD

This Indian Standard (Part 1) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Sieves, Sieving and Other Sizing Methods Sectional Committee had been approved by the Civil Engineering Division Council.

Gravitational sedimentation particle size analysis methods are based on the settling velocity, under a gravitational field of particles in a liquid. This method is widely used for determining size distribution of many particulate materials. Typically, the gravitational methods apply to samples in the 0.5 µm to 100 µm size range and where the sedimentation condition for a Reynolds number < 0.25 is satisfied.

The choice of the most suitable method for determination of size distribution depends upon factors like:

a) the purpose for which the analysis is required,
b) the size range and density of particles,
c) the important properties of powder,
d) the amount available for test, and
e) the method by which the gross sample has been collected.

Due to the inherent characteristics of the different materials and the factor stated above, it is not possible to apply a single method of size analysis. This standard recommends procedures that may be applied in the majority of cases. The purpose of this standard is to provide uniformity in procedure for any gravitational method selected to facilitate comparisons of size analysis data.

This standard was first published in 1969 with the title 'Liquid sedimentation methods for determination of particle size of powders'. In this revision, to incorporate the latest advancement in gravitational liquid sedimentation methods for determining size distribution of particulate materials, the gravitational liquid sedimentation method has been grouped into four parts. This standard (Part 1) covers general principles and guidelines. The other parts in the series are:

Part 2 Fixed pipette method
Part 3 X-ray gravitational technique
Part 4 Balance method

In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

ISO 13317 (Part 1) Determination of particle size distribution by gravitational liquid sedimentation methods: Part 1 General principles and guidelines

ISO 13317 (Part 2) Determination of particle size distribution by gravitational liquid sedimentation methods: Part 2 Fixed pipette method

ISO 13317 (Part 3) Determination of particle size distribution by gravitational liquid sedimentation methods: Part 3 X-ray gravitational technique

ISO 13317 (Part 4) Determination of particle size distribution by gravitational liquid sedimentation methods: Part 4 Balance method

The composition of the Committee responsible for the formulation of this standard is given in Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DETERMINATION OF PARTICLE SIZE DISTRIBUTION BY GRAVITATIONAL LIQUID SEDIMENTATION METHODS

PART 1 GENERAL PRINCIPLE AND GUIDELINES

(First Revision)

1 SCOPE

1.1 This standard covers general principles and guidelines for determining the particle size distributions of particulate materials, typically in the size range of 0.5 µm to 100 µm, by gravitational sedimentation in a liquid.

1.2 The methods of determining the particle size distribution described in this standard are applicable to slurries or to particulate materials which can be dispersed in liquids. A positive density difference between the discrete and continuous phases is necessary, although gravitational photo sedimentation can be used for emulsions where the droplets are less dense than the liquid in which they are dispersed. Particles should not undergo any physical or chemical change in the suspending liquid. The usual precautions need to be taken with hazardous material, and explosion proof analysers are required when examining volatile liquids with a low flash point.

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard:

IS No.	*Title*
IS 33 : 1992	Inorganic pigments and extenders for paints — Methods of sampling and test (*third revision*)
IS 4124 : 1981	Glossary of terms relating to powders (*first revision*)
IS 4730 : 1994	Method for determination of density of liquids (*first revision*)
IS 4879 : 1994	Method of sub-division of gross sample of powder used for determination of particle size
IS 5282	Determination of particle size distribution by gravitational liquid sedimetation methods:
(Part 2) : 2023	Fixed pipette method (*first revision*)
(Part 3) : 2023	X-ray gravitational technique (*first revision*)
(Part 4) : 2023	Balance method (*first revision*)
IS 8883	Methods of sampling:
(Part 1) : 2005	Chemical and chemical product (*first revision*)
(Part 2)	General requirements and precautions Sampling equipment,
(Sec 1) : 1978	For solids
(Sec 2) : 1978	For liquids
(Sec 3) : 1978	For gases
IS 18100 : 2022	Sample preparation —— Dispersing procedures for powders in liquids

3 TERMS AND DEFINITIONS

3.1 For the purpose of this standard, the definitions given in IS 4124 and the following shall apply:

3.1.1 *Analysis Sample* — The portion of the laboratory sample which is used in the size analysis apparatus.

3.1.2 *Closed Pores* — Cavities that are closed off by surrounding solid and are inaccessible to the external surface.

3.1.3 *Effective Particle Density* — Particle mass divided by the volume of liquid it displaces.

3.1.4 *Laboratory Sample* — The portion of the gross sample which is delivered to the laboratory for the determination of particle size distribution.

3.1.5 *Open Pores* — Cavities that are connected to the external surface of the particle either directly or via one another.

3.1.6 *Oversize* — Portion of the charge which has not passed through the apertures of a stated sieve.

3.1.7 *Stokes Diameter* — Equivalent spherical diameter of the particle that has the same density and terminal settling velocity as the real particle in the same liquid under creeping flow conditions.

3.1.8 *Terminal Settling Velocity* — Velocity of a particle through a still liquid at which the force due to gravity on the particle is balanced by the drag exerted by the liquid.

3.1.9 *True Particle Density* — Particle mass divided by the volume it would occupy, excluding all pores, closed or open, and surface fissures.

NOTE — True particle density is sometimes referred to as the absolute particle density.

3.1.10 *Undersize* — Portion of the charge which has passed through the apertures of a stated sieve.

4 PRINCIPLES

4.1 General

Gravitational sedimentation methods are based on the settling velocity, under a gravitational field, of particles in a liquid. The relationship between settling velocity and particle size reduces to the Stokes Equation (1) at low Reynolds numbers. The Reynolds number should not exceed 0.25 if the inaccuracy in determining the value of Stokes diameter is not to exceed 3 percent.

Stokesian sedimentation analyses depend on the applicability of Stokes law. This law defines the relationship between particle size and the change in height (within the suspending fluid) of the particle as a function of the time that the particle has fallen after reaching its terminal velocity.

$$h_{\text{fall}} = \frac{(\rho_{\text{s}} - \rho_{\text{l}}) g {x_{\text{St}}}^2 t}{18\eta} \qquad \dots (1)$$

Note that h_{fall} is defined so that it increases as the particle falls to lower positions in the sedimentation vessel. This equation may be expressed such that the Stokesian diameter of the particle may be inferred from the distance it has fallen in a given time, *t*.

$$x_{\text{St}} = \sqrt{\frac{18\eta\, h_{\text{fall}}}{(\rho_{\text{s}} - \rho_{\text{l}}) g t}} \qquad \dots (2)$$

where

x_{St} = Stokes diameter in m or µm
ρ_{s} = Effective particle density in kg.m^{-3} or g.cm^{-3}
ρ_{l} = Liquid density in kg.m^{-3} or g.cm^{-3}
g = Acceleration due to gravity in m.s^{-2}
t = Sedimentation time in s

Sedimentation techniques may be classified as either incremental or cumulative. Incremental methods are used to determine the solids concentration (or suspension density) of a thin layer at a known height and time. Cumulative methods are used to determine the rate at which solids settle from the suspension. In both methods, the powder may be introduced either as a thin layer on top of a column of liquid (the line-start technique) or uniformly dispersed at the start of the analysis (the homogeneous technique). The cumulative method is not part of this standard. The incremental homogeneous technique is more often used in gravitational sedimentation (*see* Fig. 1) and is described in this standard. The line-start technique is more applicable to centrifugal sedimentation.

4.2 Calculation of Particle Size

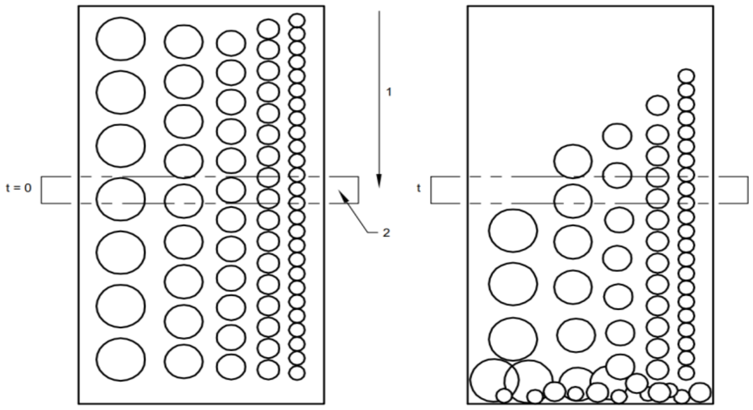
Stokes diameters are calculated according to Equation (2).

4.3 Calculation of Cumulative Mass Percentage

The cumulative mass percentage according to the particle concentration gradient in the gravitational pipette method and in the gravitational X-ray method shall be determined according to IS 5282 (Part 2) and IS 5282 (Part 3), respectively.

4.4 Effect of Measurement Zone Height on Resolution

Effect of measurement zone height on resolution is given in Annex A.



Key

t Time
1 Settling height
2 Measurement zone

FIG. 1 HOMOGENEOUS, INCREMENTAL, GRAVITATIONAL SEDIMENTATION

5 PARTICLE SIZE, SHAPE AND POROSITY LIMITATIONS

5.1 Upper Size Limit

Stokes equation predicts that the terminal settling velocity that a particle will reach in a gravitational field is:

$$v = \frac{{x_{\mathrm{St}}}^2}{K_1} \qquad \dots (3)$$

where

$$K_1 = \frac{18\eta}{(\rho_{\mathrm{s}} - \rho_{\mathrm{l}})\, g} \qquad \dots (4)$$

is expressed to solve the Stokesian diameter of the particle:

$$x_{\mathrm{St}} = \sqrt{K_1\, v} \qquad \dots (5)$$

Since the terminal settling velocity is constant and attained quickly, $h_{\mathrm{fall}} = v.\ t_{\mathrm{fall}}$ the particle diameter can be estimated from the distance the particle falls during a given time:

$$x_{\mathrm{St}} = \sqrt{\frac{K_1 h_{\mathrm{fall}}}{t_{\mathrm{fall}}}} \qquad \dots (6)$$

Upper size limit is defined by the largest particle having the terminal settling velocity, which satisfies the condition $Re < 0.25$. The Reynolds number is the ratio of inertial to viscous forces on the settling particle and is defined by the following equation:

$$Re = \frac{\rho_1\, v\, x_{\mathrm{St}}}{\eta} \qquad \dots (7)$$

Stokes equation is valid only under conditions of creeping (laminar) flow, for which the Reynolds number is less than 0.1 (*see* Annex B). Its predictions are increasingly inaccurate at higher Reynolds numbers. The inaccuracy in determining x_{St} from v is 3 percent at a Reynolds number of 0.25 beyond this, Stokes law does not provide a good estimate of particle size based on sedimentation velocity. Substituting 0.25 for Re in Equation (7), solving for v, and substituting in Equation (5) yields the recommended upper size limit of validity for the gravitational sedimentation method as:

$$x_{\mathrm{St.U}} = \sqrt[3]{\frac{0.25\, K_1\, \eta}{\rho_{\mathrm{l}}}} \qquad \dots (8)$$

where

v = Terminal settling velocity in m.s^{-1} or µm.s^{-1}
K_1 = Grouped parameter in m.s
Re = Reynolds number
$x_{\mathrm{St.U}}$ = Upper Stokes diameter in m or µm

Example:

A gravitational sedimentation measurement is carried out at 293.15 K using solid quartz spheres (ρ_s = 2 650 kgm^{-3}) in 1-propanol (ρ_{l} = 840 kg.m^{-3} and η = 2.256 mPa.s). From Equation (4), $K_1 = 2.24 \times 10^{-6}$ m.s, and from Equation (8), the maximum particle size for which Stokes law may be used (with an error of less than 3 percent) is $x_{\mathrm{St.U}}$ = 116 µm, for which v = 6.03 mm.s^{-1}.

5.2 Lower Size Limit

The lower size limit to which gravitational sedimentation methods can be applied is controlled by temperature variation, causing circulatory currents in the suspension, by flocculation of particles during the progress of sedimentation and by diffusion or Brownian motion of the very small particles.

Note that charged particles in weak electrolytes have associated with them an electrical double layer. When these particles settle, the double layer is distorted with the result that an electrical field is set up which opposes motion. These electro-viscous effects can be reduced by the use of non-ionic liquids, where possible. Information on the accuracy of Stokes law as a function of Reynolds number is given in Annex B.

5.2.1 *Thermal Diffusion (Brownian Motion)*

The random collisions of the molecules making up the liquid with a particle cause differences in the pressure on the particle from one part of the surface to another such that the particle is displaced (Brownian motion (*see* Annex C)). The equation which represents the statistical average change in position for a particle of diameter x along any one direction of motion in the absence of other forces (such as gravity) is:

$$\Delta h_{\mathrm{diff}} = \sqrt{\frac{K_2\, t_{\mathrm{fall}}}{{x_{\mathrm{St}}}^{5/2}}} \qquad \ldots (9)$$

where

$$K_2 = \frac{2\,k\,T}{3\,\pi\,\eta} \qquad \ldots (10)$$

Note that this is the statistical average of the changes in position in one direction for a large number of particles; some of the particles will travel farther from the starting point than this and some will travel a shorter distance than the average. If both gravity and thermal diffusion are considered, then a spherical particle that travels a distance h_{diff}, downward in time, t_{fall}, could be:

a) A particle whose vertical thermal motion averaged out to zero and whose diameter is correctly determined from equation (6);
b) A particle whose vertical thermal motion increased the vertical distance travelled and whose diameter is smaller than the value computed from Equation (6); and
c) A particle whose vertical thermal motion decreased the vertical distance travelled and whose diameter is larger than the value computed from Equation (6).

The ratio of distances travelled due to thermal motion and sedimentation is, from Equations (6) and (9):

$$f_{\mathrm{diff}} = \frac{\Delta h_{\mathrm{diff}}}{h_{\mathrm{fall}}} = K_1 \left[\frac{K_2}{{x_{\mathrm{St}}}^5 t_{\mathrm{fall}}}\right]^{1/2} \qquad \ldots (11)$$

The lower size limit Stokesian sedimentation analysis is generally taken as the size for which $f_{\mathrm{diff}} = 0.1$. Using this value and solving Equation (11) for x gives:

$$x_{\mathrm{St.L}} = \sqrt[5]{\frac{100\, K_1^2 K_2}{t_{\mathrm{fall}}}} \qquad \ldots (12)$$

where

Δh_{diff} = Statistical average positional change in one direction for large number of particles due to thermal diffusion in m or µm
K_2 = Grouped parameter in m^3.s^{-1}
k = Boltzmann constant in J.K^{-1}
T = Absolute temperature K (Kelvin)
f_{diff} = Fractional uncertainty of particle position due to thermal diffusion in dimensionless
$x_{\mathrm{St.L}}$ = Lower Stokes diameter in m or µm

Example:

> Using the same materials and temperature as the example given in **5.1**, gravitational sedimentation occurs over a period of t_{fall} 1 800 s (30 min). From Equation (10) $K_2 = 3.81 \times 10^{-19}$m^3. s^{-1} and from Equation (12), the smallest particle size for which the thermal broadening is less than 10 percent of the sedimentation distance is $x_{\mathrm{St.L}} = 0.64$ µm.

5.3 Particle Shape

At a low Reynolds number, the orientation of non-spherical particles is random, so a single particle will have a low range of settling velocities. As the Reynolds number increases, particles tend to align to give maximum drag and thus will settle at the slowest of the range of velocities possible with random orientation, so a particular particle may have a low or high velocity depending on its orientation.

5.4 Particle Porosity

It is recommended that the effective particle density be determined where possible, that is, the particle density be determined in the suspending liquid, plus dispersant that will be used in the sizing measurement. This will compensate for the presence of any closed porosity and also for open porosity to the extent that the chosen liquid penetrates the open porosity. For particles that are non-porous and of known composition, a density value may be taken from a handbook or determined experimentally. Information on the effect of open pores on the terminal velocity of spherical particles is given in Annex D.

6 TEST CONDITIONS

6.1 Temperature

The analysis temperature affects the liquid density and viscosity and, less significantly, the solid density values in the Stokes equation. Consequently, it is also important that the sample temperature be maintained within narrow limits for the duration of the analysis. It is recommended that the temperature of the sedimentation vessel be kept constant to within ± 1 K since the viscosity of some liquids can change significantly with temperature. If the temperature varies more than ± 1 K, then it is recommended that the temperature be noted at the beginning and at the end of the analysis and that the average value is used for viscosity calculation. To minimize convection currents, it is recommended that the rate of change of temperature be maintained at less than ± 0.05 $K.m^{-1}$.

The temperature of the suspension may be controlled or the agitated suspension may be left standing until temperature equilibrium is attained. Temperature control requirements increase with increasing fineness of the powder. The lower size limit is due, in part, to the longer sedimentation times required for the particles and the requirement to maintain stable conditions in the sedimenting suspension. The sedimentation vessel should provide a covered system to minimize evaporation of the upper layer of the sedimentation liquid, which would give rise to convection currents.

6.2 Concentration of Suspension

Stokes equation applies to the sedimentation of a single spherical particle settling relatively slowly in a liquid of infinite extent. These requirements are never fulfilled in a sedimentation analysis where particles are separated from each other and the vessel walls by finite distances. This provides for a mutual effect of the particles on each other and also neighbouring surfaces. In order to minimize these effects, low concentrations are recommended, for example. 0.2 percent volume concentration. If the recommended maximum concentration has to be exceeded, then analyses should be carried out at two or more concentrations in order to determine if the concentration effects are negligible. Wall-to-wall distances should be at least 5 mm to reduce wall effects to an acceptable level.

6.3 Sedimentation Vessel

The vessel shall be vertical to avoid convection and be free from vibration to avoid disturbances on particle settling. Unstable conditions have been attributed to the walls of the sedimentation vessel, or elements (for example, stirrer) within the walls, being slightly out of vertical. Even for vertical elements, there is an inherent instability because the return flow of liquid displaced by the sedimenting particles tends to be along vertically inclined surfaces. The vessel shall be vertical, otherwise convection currents are set up, flowing up the walls that are sheltered from sedimenting particles. These currents produce errors in the estimated size distribution.

6.4 Transient Flow

The time required for a particle to reach its steady (terminal) velocity is negligible, but excessively short sedimentation times should be avoided. Prior to particles attaining their terminal velocity, there are other local velocity fluctuations due to the sudden cessation of the initial agitation. These transient eddies and flows shall be allowed to subside.

7 SAMPLING

Controlled sampling is a necessary condition for obtaining analysis sample results for sedimentation tests. The sample shall be taken according to IS 8883. The sample division shall be according to the guidelines given in IS 4879.

8 PREPARATION FOR A SEDIMENTATION ANALYSIS

8.1 Density of Liquid and Particles

The density of the liquid at the measuring temperature shall be determined in accordance with IS 4730 and the density of the particles in accordance with IS 33.

8.2 Removal of Oversize Particles

As indicated in **5.1**, for a given liquid the largest particle of the analysis sample should not exceed a certain value. The size distribution of the oversize fraction can be found by repeating the analysis using a more viscous liquid such that the coarsest particle present in the sample is less than that given by the limiting Reynolds number.

Oversize particles can be removed, by wet or dry sieving. Wet sieving should be carried out with the sedimentation liquid. Record the percentage residue on the sieve or sieves. The data from the sieve analysis, carried out on a separate portion of the laboratory sample, should be combined with the sedimentation data.

8.3 Selection of Suspending Liquid

Many powders may not always form a good dispersion in the suspending liquid alone. A suitable dispersing agent may be necessary to inhibit the formation of flocs or agglomerates during sedimentation. This agent may be incorporated in the suspending liquid or added directly to the powder. The suspending liquid shall have negligible reactivity with the sample and shall satisfy the following criteria:

a) The liquid shall have a sufficiently different density from that of the test powder to permit use of the method;
b) The liquid shall have a viscosity to accomplish the analysis within an acceptable analysis time. It should not be too long for the finer particles and not too short for the coarsest particles;
c) The liquid shall not swell or shrink the particles. If such an effect occurs, it should be determined that the effect does not exceed 5 percent diameter; and
d) The liquid shall not cause the sample to go into solution. If such an effect occurs, it shall not exceed 5 g powder per 1 kg of liquid.

8.4 Dispersion of Sample

If the particles do not readily wet in the liquid or if they form flocs under quiescent conditions, then a dispersing agent should be added to the system as per IS 18100 ‘Sample preparation — Dispersing procedures for powders in liquid’.

9 TESTS IN DUPLICATE AND VALIDATION

9.1 Tests in Duplicate

Perform tests in duplicate on analysis samples taken from the same laboratory sample. The results for duplicate analyses should normally differ less than 2 percent for the proportions of mass at the same stokes diameter. This figure would be expected to hold for the certified reference materials. A wider tolerance may be required for other sample types with narrower distributions.

9.2 Validation

The checking at regular intervals of both operator procedure and instrument performance is essential to validate the test results. Primary validation can be made with any suitable certified reference material. The total measurement procedure is examined when the reference material is analysed, including the sampling, the sample dispersion, the measurement and the subsequent calculation. The validation procedure will meet the standard if the mean value of the x_{10}, the x_{50} and x_{90} obtained from three independent measurements lies within the certified range of values of the reference material. It is recommended to use certified reference materials for validation. A record of all validation activities shall be maintained.

10 REPORTING OF RESULTS

The data shall be presented in graphical or graphical and tabular form. Results will typically be presented as Stokes diameter versus cumulative distribution by mass reported to the nearest 0.1 percent. In the case of a plot, the diameters will be placed on the abscissa and the cumulative mass percentage on the ordinate. The report shall state the following:

a) Instrument type used;
b) Test sample identification;
c) The powder, its density and mass, where applicable;
d) The suspending liquid, its temperature, density, viscosity and volume, where applicable;
e) The dispersing agent and its concentration;
f) The method of dispersion of the suspension, including the time of dispersion;
g) The method of treatment of the test sample (drying, de-agglomeration), if any; and
h) Any other information which could have an influence on the results.

ANNEX A

(*Clause* 4.4)

THE EFFECT OF MEASUREMENT ZONE HEIGHT

The detector in a sedimentation analyser measures the particle concentration in a thin horizontal slice of a column of sedimenting particles. This concentration does not distinguish between the large particles and the smaller ones, so the thicker the measurement window the lower the resolution. The zone-height-limited resolution (P_{zone}) can be defined as the ratio of the diameters for particles just exiting the bottom of the measurement zone ($x_{\text{St.h}}$) to the difference between that diameter and that for particles just entering the top ($x_{\text{St.h-}\Delta\text{h}}$) of the measurement zone.

$$P_{\text{zone}} = \frac{x_{\text{St}.h}}{x_{\text{St.h}} - x_{\text{St.h}-\Delta\text{h}}} \quad \ldots (13)$$

Substituting for x_{St} from Equation (6) and noting that the time at which the concentration measurement is made is the same at the top and bottom of the slice, gives:

$$P_{\text{zone}} = \frac{\sqrt{h}}{\sqrt{h} - \sqrt{h - \Delta h_{\text{zone}}}} \quad \ldots (14)$$

The resolution ratio acceptable for Stokesian analysis is generally taken as 14. Equation (14) can be rearranged to determine the distance to the bottom of the measurement zone as a function of the measurement slice thickness and the resolution:

$$h_{\text{zone},P} = \frac{h_{\text{zone}} P^2}{2P - 1} \quad \ldots (15)$$

The minimum distance for acceptable resolution ($P_{\text{min}} = 14$) in the gravitational sedimentation method is the h_{zone}. $P_{\text{min}} = 7.26\ \Delta h_{\text{zone}}$. This Equation is valid whether the method uses a fixed position for the measurement zone or scans the zone upward at an ever-decreasing rate (a 'hyperbolic scan') so that the position of the detector zone varies with time as:

$$h_{\text{zone}} = \frac{K_{\text{scan}}}{t} \quad \ldots (16)$$

The time at which the detector passes $h_{\text{zone}.P_{\text{min}}}$ is the limit of scan time for which resolution is acceptable, so:

$$t_{\text{limit}} = \frac{K_{\text{scan}}}{K_{\text{zone}.p_{\text{min}}}} = \frac{K_{\text{scan}}}{7.26\ \Delta h_{\text{zone}}} \quad \ldots (17)$$

Substituting this as t_{fall} in Equation (12) yields the lower limit of particle size for which gravitational measurement is acceptable for instruments using the hyperbolic scan method:

$$x_{\text{St.L}.P\text{min}} = \sqrt[5]{\frac{726\ K_1^2 K_2 \Delta h_{\text{zone}}}{K_{\text{scan}}}} \quad \ldots (18)$$

where

P_{zone} = Zone-height-limited resolution
$x_{\text{St.h}}$ = Particle diameter exiting measurement zone in m or µm
h = It is the height (to the bottom of the measurement zone)
Δh = It is the thickness of the measurement zone
Δh_{zone} = Thickness of measurement zone in m or µm
P = Resolution ratio
K_{scan} = Hyperbolic scan constant in m.s
P_{min} = Minimum acceptable resolution
$K_{\text{zone}.P\text{min}}$= Minimum settling distance for acceptable resolution, Pmin in m or µm

Example:

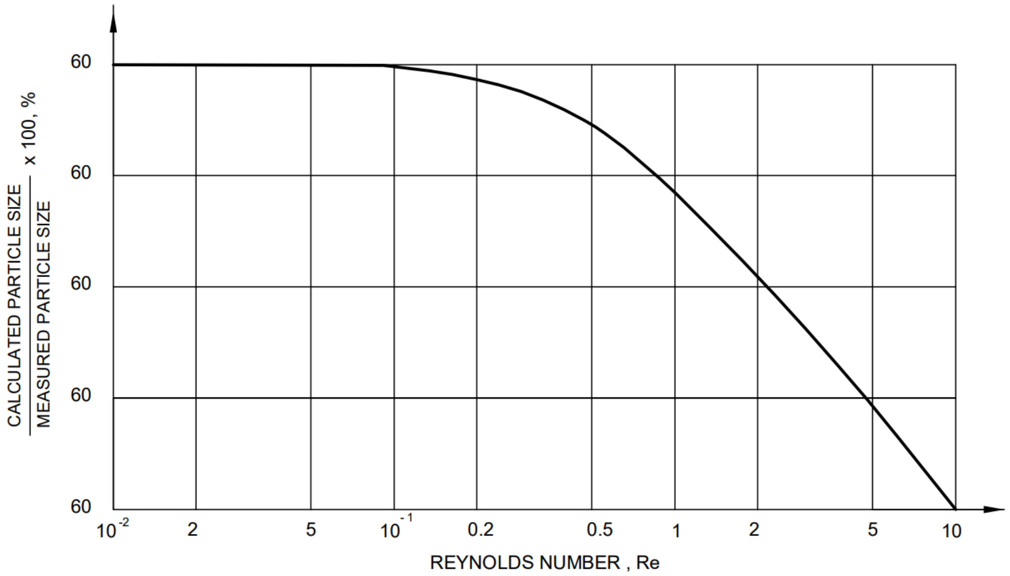
A gravitational sedimentation measurement is carried out at 293.15 K using solid quartz spheres (ρ_s = 2 650 kg. m^{-3}) in 1-propanol (ρ_{l} = 804 kg. m^{-3} and η = 2.256 mPa. s). Measurement zone of height $\Delta h_{\text{zone}} = 100\ \mu\text{m}$ is scanned upward at an ever-decreasing rate with a hyperbolic scan constant $K_{\text{scan}} = 2$ m. s. From Equations (15) and (17), the height at which the resolution drops below 14 is $h_{\text{zone}.P_{\text{min}}} = 726\ \mu\text{m}$ and the time the detector reaches this point is $t_{\text{limit}} = 2755$ s (49.3 min). From Equation (18), the minimum particle size for which resolution and thermal broadening are acceptable is $x_{\text{St.L}.P_{\text{min}}} = 0.58\ \mu\text{m}$.

ANNEX B

(*Clauses* 5.1 *and* 5.2)

ACCURACY OF STOKES LAW AS A FUNCTION OF REYNOLDS NUMBER

In Fig. 2, the calculated particle size, making the assuming that Stokes law applies, is ratioed to the measured particle size for a range of Reynolds numbers. At a low Reynolds number, the particles settle in random orientation. At a higher Reynolds number, there is an increasing tendency for the particles to orientate to give maximum resistance to motion. Stokes law underestimates the drag coefficient as the Reynolds number increases. Thus the measured size will be larger than that estimated by using Stokes law. It would be necessary to apply diameter corrections at a higher Reynolds number, otherwise the determined diameter will vary with fluid density or viscosity. The gravitational methods are recommended to apply to samples where the sedimentation condition for a Reynolds number less than 0.25 is satisfied for the largest size of particle in the test portion.



.

FIG. 2 ACCURACY OF STOKES LAW AS A FUNCTION OF REYNOLDS NUMBER

]

ANNEX C

(*Clause* 5.2.1)

PARTICLE DISPLACEMENT DUE TO BROWNIAN MOTION

Particle displacement due to brownian motion shows particle size (on the $x-$ axis) versus the ratio of Brownian motion displacement to total particle displacement (on the $y-$ axis). This is given for a range of particle-liquid density differences, at one minute after sedimentation has commenced and enables the potential error due to Brownian motion for a given particle size to be estimated for a given sedimentation time. The particle size of interest is selected and the time axis is extended to the desired time of analysis. The vertical intercept from this point to the appropriate density difference curve permits the potential error due to Brownian motion to be determined on the $y-$ axis.

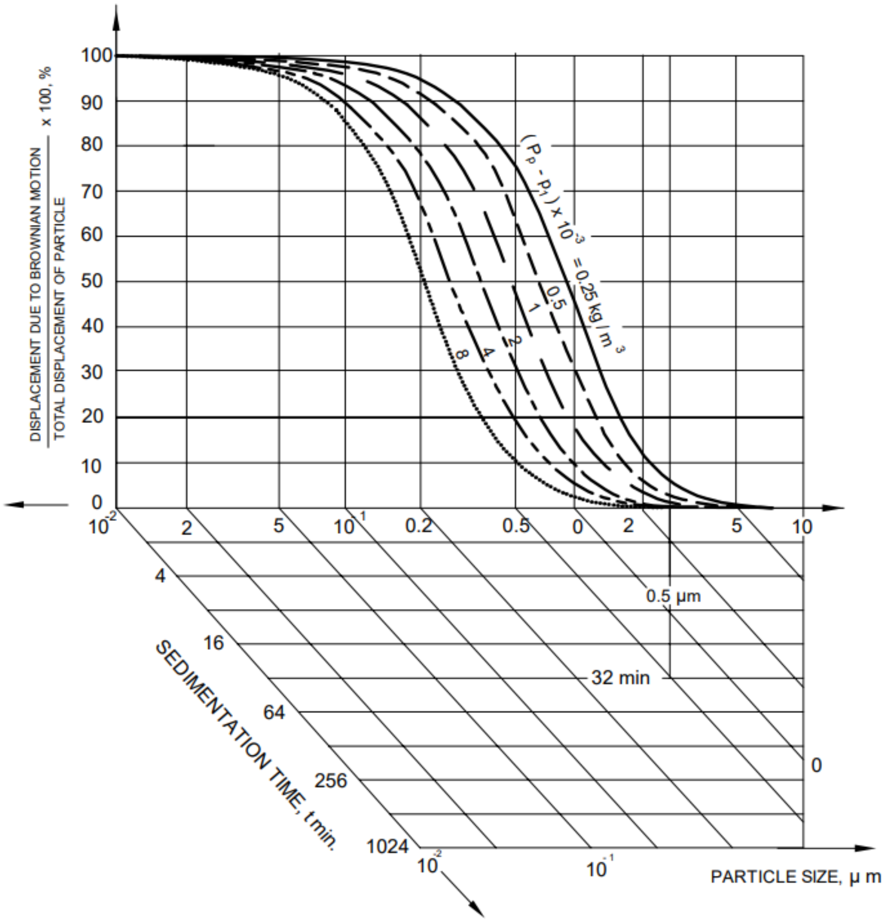


FIG. 3 PARTICLE DISPLACEMENT DUE TO BROWNIAN MOTION

ANNEX D

(*Clause* 5.4)

EFFECT OF OPEN PORES ON THE TERMINAL VELOCITY OF SPHERICAL PARTICLES

Particles may include porosity. This porosity can consist of open pores, where cavities are connected to the external surface of the particle, or closed pores, where cavities remain inaccessible to the external surface, or a combination of both porosity types. If all the pores are open, then it is possible to evaluate the particle size measurement error due to the presence of this open porosity. When a spherical particle of diameter, x, true density, ρ_p, and porosity, ε, is filled with liquid of density, ρ_l, to the fraction of f and sediments in liquid of density, ρ_l, then its terminal velocity, v, can be determined as follows. Volume of the particle is given by:

$$\left(\frac{\pi}{6}\right)x^3 \qquad \dots (19)$$

the result being that volume of the particle corresponds to the 'envelope particle volume' that is, including the volume contributed by the open porosity. The mass of particle is given by:

$$\left(\frac{\pi}{6}\right)x^3\left[(1-\ \varepsilon)\rho_\mathrm{p}+\rho_\mathrm{l}\varepsilon\, f\right] \qquad \dots (20)$$

the result being the 'effective mass' of the particle, i.e. allowing for that mass of liquid inside the pores of the particle which during sedimentation moves wholly as if part of the particle itself.

Gravitational force acting on particles is given by:

$$\left(\frac{\pi}{6}\right)x^3\left\{\left[(1-\varepsilon)\rho_\mathrm{p}+\rho_\mathrm{l}\varepsilon\, f\right]-\rho_\mathrm{l}\right\}g \qquad \dots (21)$$

and the drag force acting on the particle is given by:

$$3\,\pi\,\eta\,x\,v \qquad \dots (22)$$

then terminal velocity is:

$$v=\left(\frac{x^2\{[(1-\ \varepsilon)\rho_\mathrm{p}+\rho_\mathrm{l}\varepsilon\, f]-\rho_\mathrm{l}\}g}{18\eta}\right) \qquad \dots (23)$$

In a measurement, however, this velocity is regarded as corresponding to a particle of density, ρ_p, with no porosity. This measured diameter, x', which makes no allowance for the presence of fluid-filled open porosity, can be expressed by:

$$v=\frac{x'^2\langle\rho_\mathrm{p}-\rho_\mathrm{l}\rangle g}{18\eta} \qquad \dots (24)$$

The ratio of the measured particle diameter, x', to the true particle diameter, x, may be expressed as:

$$\frac{x'}{x}=\sqrt{\frac{\langle(1-\ \varepsilon)\rho_\mathrm{p}+\rho_\mathrm{l}\varepsilon\, f\rangle-\rho_\mathrm{l}}{\rho_\mathrm{p}-\rho_\mathrm{l}}} \qquad \dots (25)$$

When the particle porosity is completely filled by sedimentation liquid ('wet' in Fig. 4) then the following relative particle size relationship holds:

$$\frac{x'}{x}=\sqrt{1-\ \varepsilon} \qquad \dots (26)$$

When no liquid enters the open porosity ("repel" in Fig. 4) then the relative particle size relationship is given by:

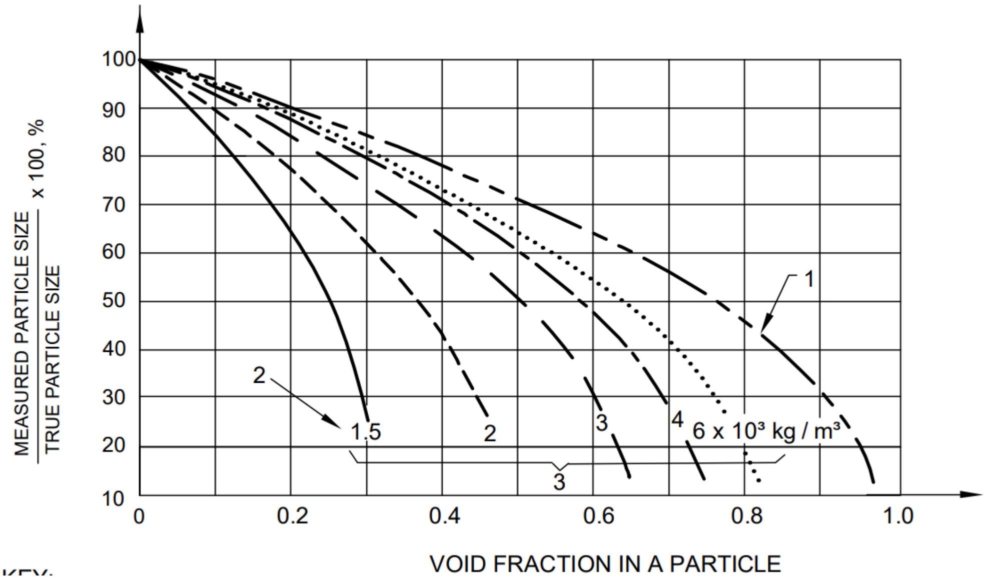
$$\frac{x'}{x}=\sqrt{1-\frac{\rho_\mathrm{p}\varepsilon}{\rho_\mathrm{p}-\rho_\mathrm{l}}} \qquad \dots (27)$$

where

ε = Particle porosity
f = Fraction of open particle porosity filled with sedimentation liquid
p = True particle density (no porosity) in kg.m^{-3} or g.cm^{-3}

The effect on the measured diameter of filling the particle void fraction, with water as the sedimentation liquid ($\rho_\mathrm{l} = 1 \times 10^3$ kg. m^3), is assessed in Fig. 4 for a range of particle densities.

Note that the particle density, ρ_p in the diagram is the 'true' density when there is no particle porosity. The curves in Fig. 4 consider the case only for the complete absence of filling, $f = 0$ (repel), and for the complete filling of open porosity, $f = 1$ (wet). Where f has some value between 0 and 1, then intermediate curves, corresponding to partial pore filling, would obtain between the 'wet' and 'repel' curves for a given particle density.



Key

1 Wet
2 Particle density
3 Repel water

FIG. 4 EFFECT OF OPEN PORES ON THE TERMINAL VELOCITY OF SPHERICAL PARTICLES

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Sieves, Sieving and Other Sizing Methods Sectional Committee, CED 55

Organization	*Representative(s)*
In Personal Capacity (*90, Savita Vihar, Vikas Marg, Delhi - 110092*)	Dr R. P. Singhal (***Chairperson***)
AIMIL Limited, New Delhi	Shri Rohitash Barua Shri Madan Kumar Sharma (*Alternate*)
Associated Soapstone Distributing Co Pvt Ltd, Jaipur	Shri Vikram Golcha Shri Dilip Jha (*Alternate*)
Cement Corporation of India Limited, New Delhi	Shrimati Saraswathi Devi Shri Anurag Kumar Saini (*Alternate*)
Central Public Works Department, New Delhi	Shri Vinayak Rai Shri Divakar Agrawal (*Alternate*)
Central Soil & Materials Research Station, New Delhi	Shri Amardeep Singh Shri B. K. Munzni (*Alternate*)
CSIR - Central Building Research Institute, Roorkee	Shri A. K. Sharma Dr Pradeep Kumar (*Alternate*)
CSIR - National Physical Laboratory, New Delhi	Dr Sanjay Yadav Dr Nita Dilawar (*Alternate*)
Haver Standard India Pvt Limited, Mumbai	Shri Deven H. Shah Shri Vikram D. Shah (*Alternate*)
Indian Bureau of Mines, Nagpur	Shri M. G. Raut Shri V. A. Sontakkey (*Alternate*)
Indian Institute of Technology, New Delhi	Representative
Industrial Wire Netting Company, New Delhi	Shri Keshav Gupta Shri Sanjay Gupta (*Alternate*)
Military Engineer Services, Engineer-in-Chief's Branch, Integrated HQ of Ministry of Defence, New Delhi	Shri P. K. Jain Shri Somesh Kumar (*Alternate*)
National Council for Cement and Building Materials, Faridabad	Shri Suresh Kumar Shri G. J. Naidu (*Alternate*)
National Mineral Development Corporation Limited, Hyderabad	Shri S. K. Sharma Shri G. V. Rao (*Alternate*)
National Test House, Kolkata	Shri Vivek Sharma Shrimati Maymole Boben (*Alternate*)
Steel Authority of India Ltd, Kolkata	Shri Bipin Kumar Giri Shri Sibabrata Basak (*Alternate*)
Ultratech Cement Ltd, Mumbai	Shri Dinesh Chandran K. P. Shri Devendra Pandey (*Alternate*)

Organization	*Representative(s)*
In Personal Capacity (*B- 129, F-2, Ramprastha Colony, Ghaziabad - 201011*)	SHRI VIRENDRA BABU
In Personal Capacity (*Indian Institute of Technology, Hauz Khas, New Delhi - 110016*)	PROF B. PITCHUMANI
BIS Directorate General	SHRI ARUNKUMAR S., SCIENTIST 'E'/DIRECTOR AND HEAD (CIVIL ENGINEERING) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
DR MANOJ KUMAR RAJAK
SCIENTIST 'D'/JOINT DIRECTOR
(CIVIL ENGINEERING), BIS

This Page has been Intentionally left blank

This Page has been Intentionally left blank

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Head (Publication & Sales), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: CED 55 (18396).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	2821 8093

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. CHANDIGARH. CHENNAI. COIMBATORE. DEHRADUN. DELHI. FARIDABAD. GHAZIABAD. GUWAHATI. HIMACHAL PRADESH. HUBLI. HYDERABAD. JAIPUR. JAMMU & KASHMIR. JAMSHEDPUR. KOCHI. KOLKATA. LUCKNOW. MADURAI. MUMBAI. NAGPUR. NOIDA. PANIPAT. PATNA. PUNE. RAIPUR. RAJKOT. SURAT. VISAKHAPATNAM.

Published by BIS, New Delhi